

THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re: the application of

Vance BERGERON et al

Application No.: 09/698,479

Examiner: William Cheung K.

Filed: October 30, 2000

Art Unit: 1615

For: POLYMERS, COMPOSITIONS AND METHODS OF USE FOR FOAMS LAUNDRY  
DETERGENTS SHOWER RINSES AND COAGULANTS

**RULE 123 DECLARATION**

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

I, Mark R. Sivik, declare as follows.

1. I, Mark R. Sivik received my Bachelor of Science in chemistry at Cleveland State University in 1987 and a Ph.D. in organometallic chemistry from The Ohio State University in 1992. Since 1992, I have been employed by the Procter & Gamble Company as a scientist in the Household Care Business Unit wherein my focus is in the area of developing new chemical technologies for the business unit's consumer products. I am a named co-inventor of the presently claimed application.

2. I am familiar with the prosecution of the above-identified patent application and the pending Final Office Action mailed August 26, 2006. In this Office action Claims 1-13, 16-18 and 42 are rejected under 35 USC §102 as being anticipated, or in the alternative under 35 USC 103 as being unpatentable, in view of US 4,542,175 (Fink et al.).

3. Upon reviewing the disclosure of Fink et al. it is my opinion that the materials taught by Fink et al. are not readily soluble in a water solution such as the described polymers of the present application. For example, use of a butyl acrylate monomer as demonstrated in Examples 1-7c of Fink et al. in Col. 10, will make the polymer of the present application too hydrophobic and therefore not effective for suds boosting properties in a water solution such as hand dishwashing compositions. Further examples in Fink et al., do not use butyl acrylate, but do use ethyl acrylate and methacrylate. It is also my opinion that these polymer compositions

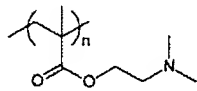
having ethyl acrylate and methacrylate will also be too hydrophobic for use in the present application.

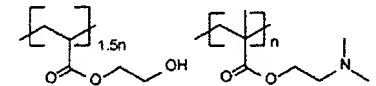
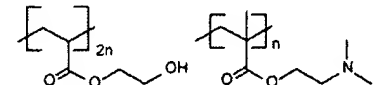
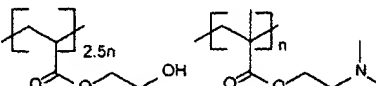
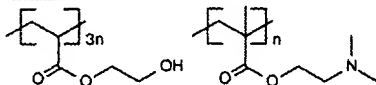
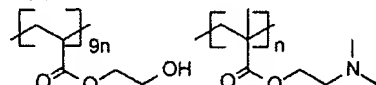
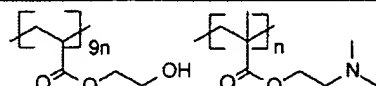
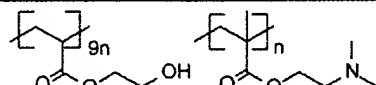
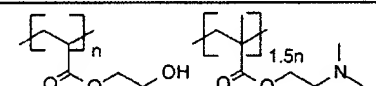
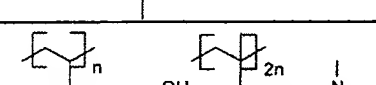
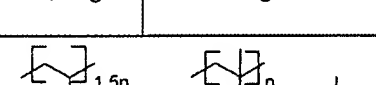
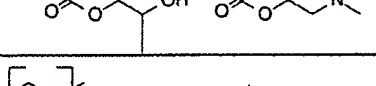
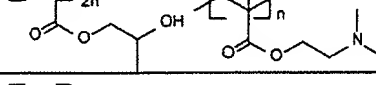
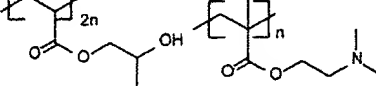
4. It is further my opinion that Fink et al. fails to discuss homopolymers or copolymers of dimethylaminoethyl methacrylate in order for one of skill in the art to arrive at the claimed polymers of the present invention that are used in cleaning and do not thicken the formulation in a significant fashion.

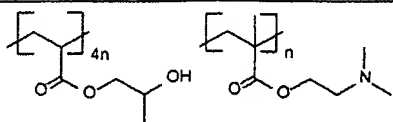
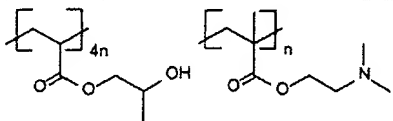
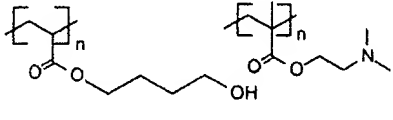
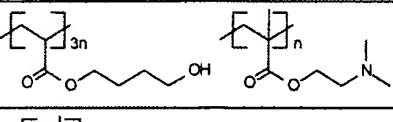
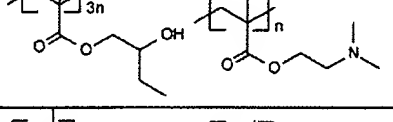
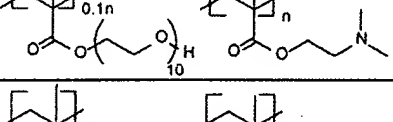
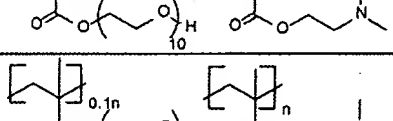
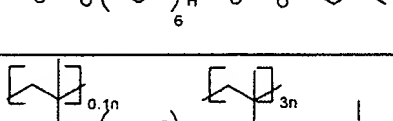
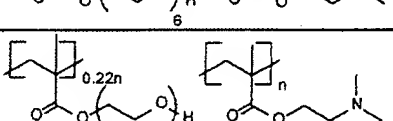
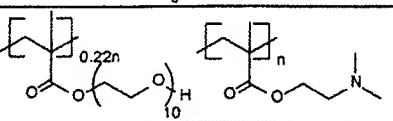
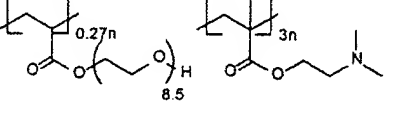

5. In contrast, the present invention employs the claimed co- and terpolymers. The charge density of the claimed polymer is critical for suds stabilization through favorable polymer/soil interactions that prevent soil antifoam effects, but as wash pH varies, so can the cationic charge density which can cause negative interactions with any anionic surfactant that is present, leading to a loss of suds. To reduce the cationic charge and pH dependence of the polymer/soil interaction for the polymer, several alternative mechanisms, in conjunction with cationic charge, to increase polymer/soil interactions may be used. They are: 1) lower the overall charge density to minimize cationic charge and pH dependence of the polymer/soil interaction via the introduction of non-charged co-monomers with dimethylaminoethyl methacrylate and 2) increase hydrophobicity to drive the polymer/soil interaction away from electrostatic and closer to hydrophobic interaction via more hydrophobic non-charged co-monomers with dimethylaminoethyl.

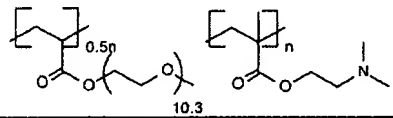
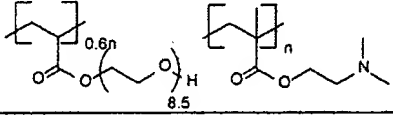
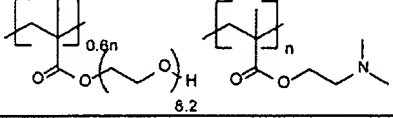
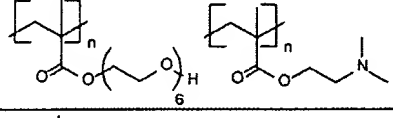
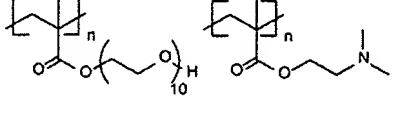
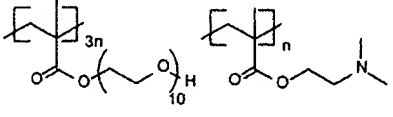
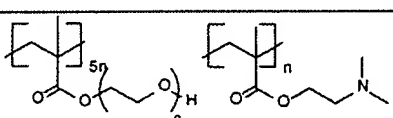
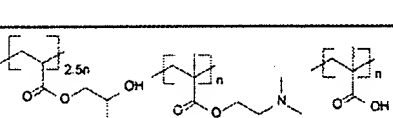
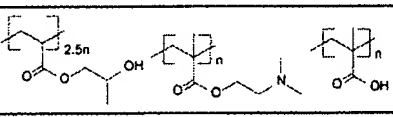
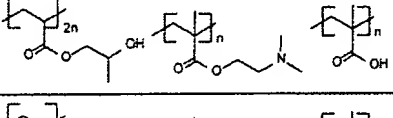
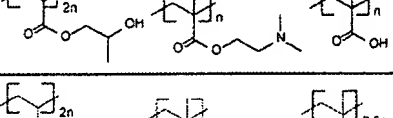
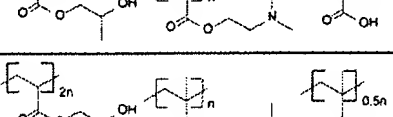
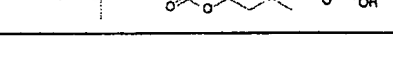
6. Co-monomers selected such as hydroxy ethyl acrylate, hydroxy propyl acrylate, hydroxy butyl acrylate, poly(ethylene glycol) acrylate and acrylic acid were added to dimethylaminoethyl methacrylate as shown in Table 1.

**Table 1**

Sample	Structure	MW	pKa	formula table	Performance Results Hand dishwashing composition, 15 gpg, 115°F, 5ppm (unless stated otherwise)	
					greasy soil	proteinaceous soil
1.	 DMAM (comparative)	160 K	7.7	Yes	109/111	87/86

2.		51K	--	Yes	120/118	82/90
3.		36K	7.8	Yes	119/115	91/95
4.		32K	--	Yes	116/115	98/100
5.		670 K	7.7	Yes	117/114	96/92
6.		78K	--	X	--	--
7.		45K	--	X	--	--
8.		17K	--	X	--	--
9.		13K	7.5	Yes	120/118	--
10.		14K	7.6	Yes	120/118	--
11.		26K	7.4	Yes	113/110	--
12.		11K	7.5	Yes	110/110	--
13.		278 K	7.5	Yes	123/119	98/98
14.		280 K	7.4	C	115/117	108/107

15.		--	7.6	n/a	108/105	102/105
16.		--	--	n/a	113/111	104/105
17.		--	--	Yes	106/107	78/84
18.		--	7.0	n/a	116/111	102/98
19.		--	7.0	n/a	INSOLUBLE	--
20.		31K	7.7	Yes	129/130	89/92
21.		26K	7.7	n/a	115/115	83/88
22.		33K	--	Yes	127/119 120/114	91/88 81/88
23.		23K	--	n/a	117/114	89/92
24.		25K	--	n/a	122/114	88/92
25.		62K	7.8	n/a	125/125	84/92
26		121 K	--	n/a	118/110	85/88

27.		30K	--	n/a	106/105	94/96
28.		31K	--	n/a	108/105	97/96
29.		36K	--	n/a	117/114	93/92
30.		59K	--	n/a	111/110	99/96
31.		200 K	7.0	n/a	107/110	108/104
32.		195 K	7.1	n/a	106/110	105/104
33.		188 K	--	n/a	112/115	100/100
34.		--	9.2	n/a	101/96	99/95
35.		--	9.4	n/a	100/96	100/95
36.		--	9.3	n/a	99/96	101/100
37.		--	9.3	n/a	99/96	103/100
38.		--	8.4	n/a	114/104	104/100
39.		--	8.4	n/a	107/100	98/100

X : not formulatable, C: cloudy, H: hazy

7. The performance of these polymers for suds boosting where tested in a hand dishwashing composition (see below in Table 2) for suds volume and suds mileage against the same hand dishwashing composition (referred to below as "LDL") having 2 wt% of DMAM homopolymer. Suds volume represents that volume of suds measured both before and after the addition of the identified soil type via the Suds Cylinder Test Method. The Suds Cylinder Test Method is attached hereto as Attachment A. Suds height change is measured in the suds cylinder test. This is demonstrated in figure 1.

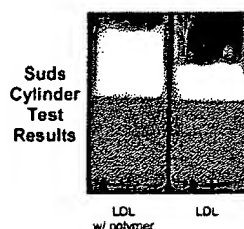


Figure 1

Suds mileage represents the number of soiled plates that can be washed with suds still being present. The Suds mileage method is attached hereto as Attachment B.

TABLE 2	
@ pH 10	Wt %
sodium C <sub>11-12</sub> ethoxylated (0.6) sulfate surfactant	29
sodium xylene sulfonate	3.0
C <sub>10-12</sub> nonionic ethoxylate surfactant	4.88
Maleic acid	3.15
Amine oxide	7.5
1,3 pentane diamine	4.88
Suds boosting polymer	2.0
water	balance

8. A homopolymer of DMAM is shown as polymer #1 in Table 1 as a comparative example for the suds volume and suds mileage testing as volume/mileage for each of the soil types listed (greasy and proteinaceous soils). Numbers reported as being larger than those reported for the homopolymer of DMAM performed better vs. the homopolymer of DMAM. Numbers reported as being smaller than those reported for the homopolymer of DMAM performed worse vs. the homopolymer of DMAM.

9. Lowering the overall charge density to minimize cationic charge and pH dependence

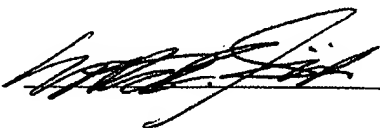
of the polymer/soil interaction via the introduction of non-charged co-monomers with dimethylaminoethyl methacrylate facilitates improved sudsings and mileage against proteinaceous soils as demonstrated by polymers 3-5, 13-16, 18, 20, 22, and 27-39. Increased hydrophobic non-charged co-monomers with dimethylaminoethyl methacrylate can be seen with the progressive increase in hydrophobicity of the co-monomers (e.g., hydroxy ethyl acrylate < hydroxy propyl acrylate < hydroxy butyl acrylate). However, too much hydrophobicity can make the polymer insoluble or slightly insoluble that causes a composition to be hazy or cloudy. This is demonstrated in polymers 6-8, 14 and 19.

10. By increasing hydrophobicity to drive the polymer/soil interaction away from electrostatic and closer to hydrophobic interaction via more hydrophobic non-charged co-monomers with dimethylaminoethyl methacrylate facilitates improved sudsings and mileage against greasy soils as demonstrated by polymers 2-5, 9-11, 13-14, 16, 18, 20-26, 29, 33, and 38.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

Date: FEBRUARY 17, 2006

By: \_\_\_\_\_

Type Name: Mark R. Sivik

## ATTACHMENT A

### Suds Cylinder Test Method (SCT)

#### **Equipment:**

Cylinder Suds Machine with 8 calibrated cylinders, lids and rubber stoppers.  
4L flask.  
500ml graduated cylinder.  
Adjustable auto pipette or syringes.  
Stirring hot plate.  
Stirring rod.

#### **Machine Design:**

The Machine has eight cylinders that have been calibrated by placing 500mls of water in each cylinder,

then placing a 5 inch ruler strip, graduated to every 0.1 inch, measuring upward on the outside of the cylinder with 0 inch at the top of the water line. The machine is to rotate at 20-21 rpm's. The speed and

time can be adjusted. The machine is preset, however, to rotate for one minute at 20-21 rpm's. To operate, turn the key to the on position. In this position the cylinders can be rotated manually by hitting the JOG button. To begin a timed series of revolutions, turn the key to the start position. This can be stopped at anytime by turning the key to the off position. All power can be turned off to the machine by hitting the EMERGENCY STOP button.

#### **Procedure:**

1. Preheat 4L deionized water to 130 -140° F.
2. Adjust water to desired hardness.
3. Adjust water to pH of 7.4. (+/- 0.1 pH units)
4. Just prior to adding water, place desired amount of product (.5cc is standard for 1X products) on the bottom of each cylinder using a 1cc syringe (or a syringe of suitable size). Do not leave products out uncovered. Evaporation of water and solvents can change the products significantly!
5. Preheat the 500ml graduated cylinder with 140° F water. Quickly measure out 500mls of water into each cylinder. Try to pour the water into the cylinders directly to the bottom without touching the sides to prevent heat loss.
6. After all the cylinders have been filled, stir each wash solution without touching the bottom with the stirring rod until all the product has dissolved.
7. Monitor the temperature of the wash solution in each cylinder. When the temp is 117° F, cap the cylinder, place rubber stopper in the cap, and attach the cylinders. Gentle swirling of the wash solution in the cylinder can help to drop the temp. But this is only suggested as a last resort for rapid cooling.
8. When all of the cylinders are ready, rotate the cylinders twice and release the pressure from each one by removing the stoppers.
9. Replace the stoppers and rotate the cylinders for two minutes. (two one minute series of revolutions)



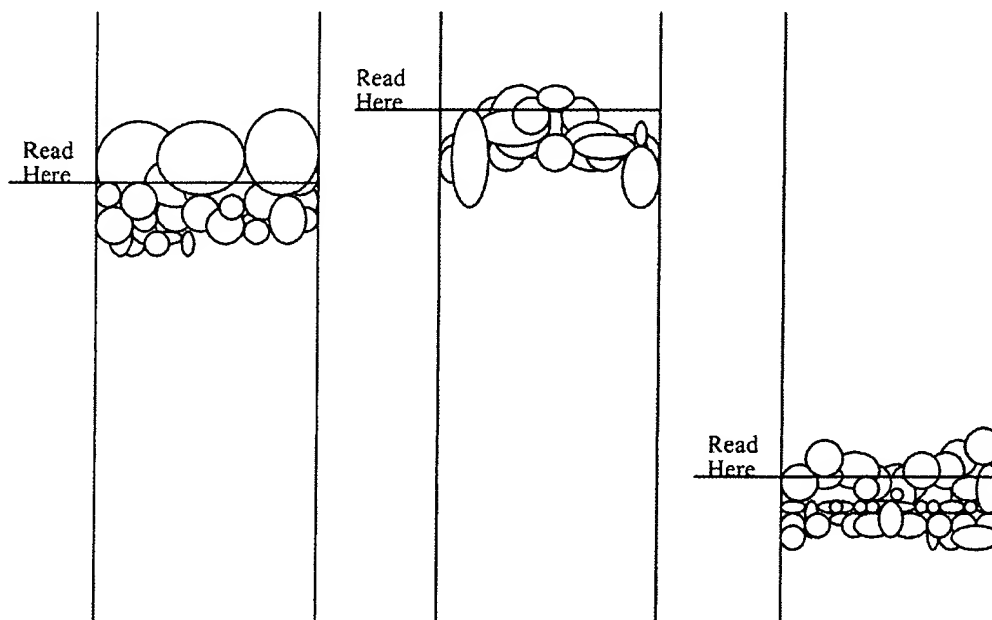
10. As quickly as possible remove stoppers, record suds height, add soil (standard is .5cc), and reseal cylinders.
11. Rotate cylinders for one minute and repeat step 10.
12. Repeat steps 10 and 11 until suds height measures less than 0.3 inches.

**Important to note:**

- Run up to four reps (standard is two). Rotate the product position in each rep.  
For example: R1: 1-2-3-4-5-6-7-8  
R2: 8-7-6-5-4-3-2-1  
Also change the position of the control from test to test.
- Should have no more than 29 soil additions. If more than 29, adjust test conditions such as product concentration or soil dosage.
- Periodically throughout the test, stir the soil and check that the soil dispenser is set correctly. Also, wipe off the tip of the pipette/syringe before each soil addition.

**Reading the Suds Heights:**

The lines across the cylinders show the best places to read the suds heights for the following examples. The rule of thumb is that if the suds are not straight across, or the top layer of suds has large airy bubbles, take the reading at a point of average between the very top and where the foam becomes level or more dense.



**Cylinder Cleaning Procedure.**

1. Dump out the soiled wash solutions.
2. Rinse cylinders with warm water.
3. Wash each cylinder, lid, and stopper with dish detergent using a scrub brush.
4. Rinse until all of the suds are gone.

5. Set stoppers aside to dry.
6. Apply a small amount of isopropanol to the inside of each cylinder and to each lid. **(If you are using plastic cylinders, you must use isopropanol. Other solvents will etch the plastic cylinders. For the sake of uniformity it is suggested to use isopropanol for the glass and plastic cylinders.)**
7. With a paper towel or a cloth, rub the isopropanol over the inside of the cylinders and the lids.
8. Rinse the cylinders and lids with de ionized water and dry.

*Procter & Gamble*  
Suds Mileage Test

## **1.0 PURPOSE**

- 1.1 The Suds Mileage Test measures the suds stability (the number of plates that can be washed until suds depletion) of household dishwashing products under conditions approximating those in the home. A plate is repeatedly soiled with an laboratory soil and washed by hand until the suds end point is reached. The data is recorded as the number of plates washed to the suds end point.
- 1.2 This procedure outlines the steps required to conduct a Suds Mileage (SM) Test method to evaluate the technical sudsing measure of light duty liquids (hand dishwashing liquids).

## **2.0 SCOPE**

- 2.1 Product concentration for the laboratory technical testing is done at typical consumer product concentration, which is 0.18% for regular dishwashing products and 0.12% for the Ultra dishwashing products. Previously, in order to evaluate more than 4 products in one test, the amount of product used was decreased to 2.5 cc per gallon (0.06% product concentration) for 1X product. Currently, it has been used for claim support at the typical consumer product concentration using a greasy soil (Representative soil). The validation of this test method was done with the typical consumer product concentration (0.18% for 1X and 0.12% for 1.5X).

## **3.0 REFERENCES**

- 3.1 None.

## **4.0 RESPONSIBILITIES**

- 4.1 It is the responsibility of the person submitting the test to supply the performance lab or product researcher with the products to be evaluated.
- 4.2 It is the responsibility of the person doing the test to be trained and qualified to run the test.
- 4.3 It is the responsibility of the person submitting the test to supply the performance lab or product researcher with a detailed request instructing them on # of products, prod. Conc., hardness etc.

## **5.0 PROCEDURE**

- 5.1 Equipment
  - 5.1.1 Round stainless steel dishpan - three gallon capacity.
  - 5.1.2 10" diameter Melmac dinner plate.
  - 5.1.3 Dishcloth (approx. 15" x 15") folded in half four times and sewn together at

the edges, with the smooth surface to the outside.

- 5.1.4 Syringes for soil and hardness dispensing.
- 5.1.5 Soil reservoir
- 5.1.6 Soil - Soil should be thoroughly remixed on the day it is used to ensure homogeneity and good flowing properties.
- 5.1.7 Suds Generator - standard "falling water" apparatus.
- 5.1.8 water -Adjusted to specific grain needed at 115°F.
- 5.1.9 Stock Water Hardness concentrate if needed
- 5.1.10 Metronome, set at 100 beats per minute.
- 5.1.11 Mechanical Counter.
- 5.1.12 Rubber gloves (optional).

## 5.2 Product Usage

- 5.2.1 Product concentration for laboratory technical testing is done at typical consumer product concentration, which is 0.18% for Regular dishwashing products and 0.12% for Ultra dishwashing products. An Ultra product has the instructions "use 1/3 less," and 0.12% is 1/3 less than 0.18%.

## 5.3 Procedure

- 5.3.1 Prepare dishpan by:
  - 5.3.1.1 Washing thoroughly with 115°F water and an LDL;
  - 5.3.1.2 Rinsing thoroughly;
  - 5.3.1.3 If the pan has not been in use in the last 15 minutes, let the dishpan sit in sink under 115°F running water for 2 minutes to bring to temperature.
- 5.3.2 Fill syringe with product (4.7 ml for Ultra or 7 ml for regular).
- 5.3.3 Prepare one gallon of 115°F water in falling water reservoir adjusted to the specific hardness needed.
- 5.3.4 Position the dishpan so that the stream of falling water will hit in the center. Dispense product into the stream of water after 5 sec. after turning on the pump to activate the falling water.
- 5.3.5 Move the dishpan to the lab bench to start washing. Depress syringe piston to deliver 4.0 ml of soil onto the plate. Place the wetted quarter-folded dishcloth onto the soil in the plate and spread the soil evenly over the entire face of the plate. (Note: Some of the soil will also be deposited in the dishcloth.) Immerse plate into the dishwater at a 15° angle from vertical (i.e., the plate front 105° from the water surface) with the bottom edge of the plate resting upon the pan bottom, halfway between the center and left edge of the pan.
- 5.3.6 With the folded dishcloth in the fingers of the hand, start washing. The

washing motion consists of one complete circular motion of the cloth from the bottom of the pan out of the water around the face and back to the original position at the bottom of the pan. The cloth should contact the plate over a palm-sized area, and about 1/10 of the soil should be removed with each stroke. When passing over the plate, use a very light pressure, sufficient, however, to squeeze some dishwater from the cloth down over the plate. Since proper soil dispersion in the water is essential, the washing action should involve the entire dishpan to some extent. This can be achieved by entering the dishpan with plate and dishcloth at an angle that moves suds and soil away from the sides toward the center and by washing with sufficient agitation to keep the entire dishpan solution well stirred.

- 5.3.7 Make ten uniformly timed washing motions in six seconds (10 beats of the metronome). After the tenth stroke, squeeze the excess water from the cloth over the back of the plate held over the center of the pan, and repeat the procedure from the addition of the soil. This constitutes the washing of one plate - six seconds to wash the plate, and six seconds to squeeze the cloth, and the soil, and spread the soil. A mechanical counter is used to register the number of plates washed. Continue this soiling/washing procedure until the suds endpoint is reached. The end point is defined as a break in the layer of suds about 3" in diameter that remains for 3 to 5 seconds.
- 5.3.8 The SMT endpoint is based on an actual consumer test (MRD84851) where panelists identified the picture which best describes the way their sink looks at the end of washing dishes. We use photo #7 as the SMT endpoint because nearly all consumers would have stopped washing dishes by sink #7, and it is a measurable endpoint. We conclude from the data that almost nobody will wash dishes in sink #8, nor would someone wash dishes in a sink that has lost all suds.
- 5.3.9 The dishpan, dishcloth, and plate should be thoroughly cleaned, as described in Paragraph #1 of this procedure, before starting the next run.
- 5.4 Record Data The suds mileage is recorded as the number of soil additions made.